Journal of Nuclear Materials 487 (2017) 343–347

Contents lists available at ScienceDirect

Journal of Nuclear Materials

journal homepage: www.elsevier.com/locate/jnucmat

A novel way to estimate the nanoindentation hardness of only-irradiated layer and its application to ion irradiated Fe-12Cr alloy

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ARTICLE INFO

Article history:
Received 3 January 2017
Received in revised form
26 January 2017
Accepted 18 February 2017
Available online 20 February 2017

Keywords:
Ion irradiation
Nanoindentation
Hardness
Fe-12Cr

ABSTRACT

While nanoindentation is a very useful tool to examine the mechanical properties of ion irradiated materials, there are some issues that should be considered in evaluating the properties of irradiated layer. In this study, in order to properly extract the hardness of only-irradiated layer from nanoindentation data, a new procedure is suggested in consideration of the geometry of indentation-induced plastic zone. By applying the procedure to an ion irradiated Fe-12Cr alloy, the reasonable results were obtained, validating its usefulness in the investigation of practical effect of irradiation on the mechanical behavior of future nuclear materials.

Energetic particle irradiation is known to seriously degrade the mechanical properties of a material [1–3]. Thus, understanding the mechanical properties in neutron irradiation environment is essential for assessing the suitability and reliability of a candidate material for nuclear applications and much research has been carried out on the related issue [4–7].

Since there are certain difficulties in exploring the effects of the neutron irradiation (e.g., it needs the long-time taken to achieve high doses, and makes the sample radioactive and thus difficult to handle), ion irradiation has been recently used as a surrogate for the neutron irradiation. However, for evaluating the irradiation-induced property change, ion irradiation has both merit and demerit in comparison of neutron irradiation; i.e., ion irradiation can produce high damage rates without residual radioactivity, whereas typical ion-irradiated layer has a limited thickness of only several μm below the irradiated surface [8]. In this regard, small-scale mechanical testing methods, especially nanoindentation test that can easily estimate the near-surface strength, have been extensively performed for estimating the mechanical behavior of ion-irradiated sample [9–13]. While nanoindentation is an useful tool for the purpose, there are still some issues that should be considered in evaluating the properties of only-irradiated layer through nanoindentation experiments, which will be introduced later. With this in mind, here we suggest a novel way to estimate the nanoindentation hardness of only-irradiated layer, and apply it to the analysis of ion irradiated Fe-12Cr alloy that is base for ferritic/martensitic steels considered as candidate materials for future reactor [7].

Fe-12Cr alloy having a chemical composition of Fe-11.9Cr-0.007C-0.021O-0.0003N (in wt%) was prepared by a vacuum induction melting using electrolytic metals. The ingot was homogenized at 1473 K for 24 h, forged, and then cold-rolled down to a thickness of 1 mm. The specimens were heat treated at a rate of 5 K/s to a recrystallization temperature for 3–5 h and then water quenched. Ion irradiation experiments were performed with a multipurpose Tandem ion accelerator at the Korea Institute of Geoscience & Mineral Resources. Before irradiation, specimens were polished electro-chemically at 18 V for 30 s in a mixture of 7% hypochlorous acid and 93% methanol for a removal of any surface damage. The specimens were irradiated with Fe^{4+} ions to three

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http://dx.doi.org/10.1016/j.jnucmat.2017.02.028
0022-3115/

different fluencies of 5.04×10^{14}, 2.80×10^{15}, and 6.72×10^{15} ions/cm^2 at room temperature (RT), and the used ion energy and beam current were 8 MeV and 200 nA, respectively. During irradiation, the backside of the aluminum sample holder was air cooled to prevent excessive heating and to keep the sample temperature below 313 K. The depth profiles of the displacement damage were calculated with the SRIM-2013 [14] based on the assumption that the value of threshold displacement energy is 40 eV [15]. The results are shown in Fig. 1 in which the maximum depth of the displacement damage is ~2.3 μm from the surface and the peak dose for the samples is 0.54, 2.69 and 6.45 displacement per atom (dpa), respectively. Hereinafter, each sample is named after its peak dose. Nanoindentation tests were performed on the ion-irradiated surfaces using a Nanoindenter-XP (formerly MTS; now Keysight, Santa Rosa, CA) with a typical Berkovich indenter. The sample is loaded to the peak load of 250 mN at a constant strain rate of 0.025 s^{-1}.

Fig. 2(a) provides representative nanoindentation load-displacement (*P*-*h*) curves of an unirradiated and three irradiated samples. In the figure, from the data set obtained under continuous stiffness measurement (CSM) module, which allows to get hardness (*H*) values continuously as a function of *h* from single nanoindentation [16], the selected *H* values for *h* of 250–2500 nm with an interval of 250 nm are exhibited. It is evident that unirradiated sample exhibits a larger peak-load displacement (h_{max}) than irradiated ones, and the h_{max} decreases as the dose increases. From the curves, hardness (*H*) values were estimated according to the Oliver-Pharr method [16], and the obtained *H* values are given as a function of *h* in Fig. 2(b). Only the *H* values taken at $h > 200$ nm are considered here, in order to avoid possible artifacts rising from the imperfect indenter tip geometry and surface roughness. Fig. 2(b) demonstrates that there is a hardening behavior after ion irradiation and it becomes more pronounced with increasing the maximum dose. The increase in *H* of irradiated samples above that of unirradiated one is more clearly visible at a lower *h*.

Despite the obvious irradiation-induced hardening phenomenon in Fig. 2(b), one should be careful in interpreting the nanoindentation results in a quantitative way since there are several issues to be considered for more accurate analysis, as Hosemann et al. [10] pointed out. One of the crucial issues is related with the plastic zone developed underneath the indenter. Since the volume of the plastic zone is in general much larger than the indented volume, considerable portion of the plastic zone volume may correspond to the unirradiated material located below irradiation layer, leading to a reasonable expectation that *H* values from nanoindentation of irradiated surfaces can be seriously affected by unirradiated material. Efforts were made to consider this and thus to estimate the *H* of only-irradiated region (H_{irr}). For example, Hosemann et al. [10] suggested a solid approach based on the rule-of-mixture to correct the errors rising from the presence of unirradiated material within the plastic zone. Assuming that plastic zone is a hemisphere with a radius of five times the indentation depth (i.e., $r_p = 5h$ where r_p is the plastic zone radius), the volume fraction of irradiated layer in the plastic zone (V_{irr}) at a given *h* was determined by simply considering the thickness of irradiated layer (that can be estimated by SRIM; e.g., ~2.3 μm in Fig. 1). Then, the H_{irr} could be estimated by a simple rule-of-mixture with the calculated volume fraction of irradiated layer within the plastic zone. Although the procedure is reasonable and appropriate H_{irr} was obtained [10], the results could be somewhat semi-quantitative estimates due to too simplified assumptions (such as $r_p = 5h$) without detailed consideration of inhomogeneous property change within the irradiated layer.

In order to complement the simplification and thus to estimate more accurate H_{irr}, a modified way is suggested as follows. It should be noted that this approach is valid only in the case that a few data of yield strength, σ_y, at different dose are available, as to be introduced. First of all, for calculating the volume of indentation-induced plastic zone, we adopted the Johnson's expanding cavity model [17] in which plastic zone produced by conical indentation is assumed to be hemispherical with a radius r_p that is given by:

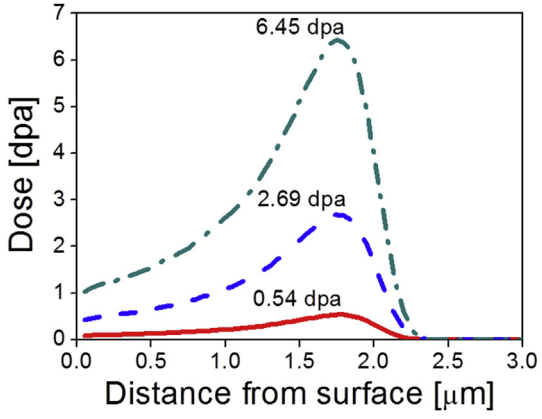
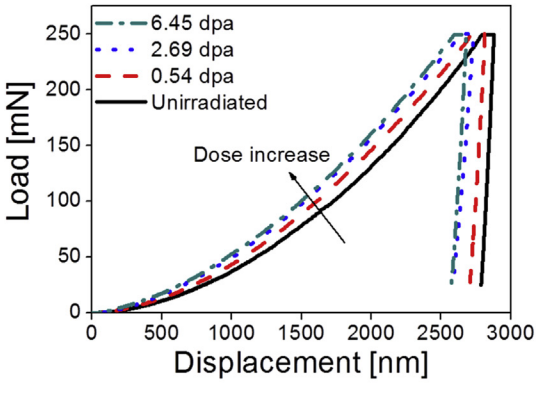
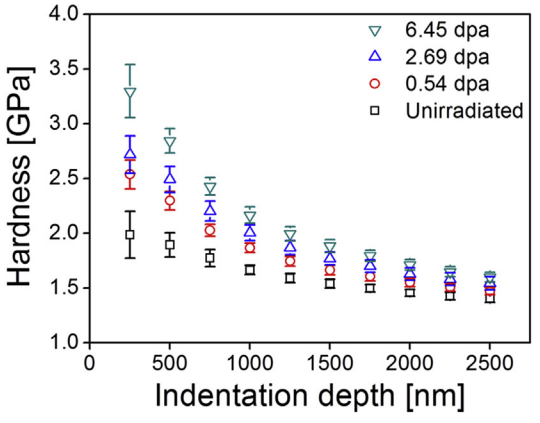


Fig. 1. Calculated results of irradiation damage vs. distance from the sample surface.



(a)



(b)

Fig. 2. Nanoindentation test results of unirradiated and irradiated samples; (a) load-displacement (*P*-*h*) curves, (b) depth-dependent hardness change.